
फॉस्फेट समृद्ध जैविक खाद — विशिष्टि

Phosphate Rich Organic Manure — Specification

ICS 65.080

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Soil Quality and Fertilizers Sectional Committee had been approved by the Food and Agriculture Division Council.

Composts are excellent source of organic carbon and are the basic food material for all the microorganisms present in the soil, but are very poor in terms of fertilizing nutrients. Keeping in view of their growing requirement for sustenance of soil health and fertility, need is also being felt to enrich them nutritionally through natural sources of nutrients. Nutritionally rich composts/manures will not only provide much needed organic matter for required biological activity, but will also help in reducing the requirements of nutrients in chemical form. Nutrients applied through the route of composts have an additional advantage of their higher use efficiency.

Phosphate rich organic manure (PROM) is nutrient rich compost, obtained by the co-composting of organic matter, including municipal solid waste with rock phosphate as natural phosphorus source. During the microbial degradation process, part of the insoluble phosphorus present in the rock phosphate gets converted into available form through the activity of microorganisms and is available to plants immediately after its application. Application of naturally occurring phosphate along with compost can be an ideal fertilizer Phosphate replacement in due course of time.

Use of farm yard manure or agro-waste in conjunction with rock phosphate is the ideal choice for PROM making, but use of municipal solid waste compost requires some degree of precaution to avoid the incorporation of heavy metals. This Indian Standard for PROM has been prepared with the aim to ensure consistent quality of PROM with adequate quantity of phosphorus in the available form along with the total phosphorus strength. This standard also provides for maximum acceptable concentrations of heavy metals to ensure that consistent usage of such inputs over a long period of time does not pose any health hazard to soil and crop productivity.

With the growing awareness for maintenance of soil health and fertility, demand is growing for organic fertilizers. Ensuring the availability of phosphorus through organic way is a recent phenomenon and is being promoted as ideal phosphorus source with high degree of use efficiency. Many industries and trading house have started producing and trading the PROM. To ensure the quality to the farmers and to maintain the standards during production process for manufacturers, standards and its testing procedures are needed.

Committee responsible for the formulation of this standard is listed in Annex L.

In the formulation of this standard, due consideration has been given to the *Fertilizer (Control) Order*, 1985 and *Legal Metrology (Packaged Commodities) Rules*, 2011 of Government of India. However, this standard is subject to restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)', The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHOSPHATE RICH ORGANIC MANURE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of sampling and tests for phosphate rich organic manure.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitutes provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard listed below:

IS No.	Title
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (fourth revision)
IS 1070 : 1992	Reagent grade water (third revision)

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Compost — Substance made of one or more unprocessed waste material of biological nature (plant and animal) and may include unprocessed

mineral material that has been altered through microbiological decomposition.

3.2 Municipal Solid Waste Compost (MSWC) — Product made from biodegradable municipal solid waste which has been altered into manure through microbiological decomposition.

3.3 Phosphate Rich Organic Manure (PROM) — Substance made of one or more unprocessed waste material of biological nature (plant and animal) and unprocessed mineral rock phosphate that has been altered through microbiological decomposition.

3.4 Rock Phosphate — Rock phosphate is a mined sedimentary rock which contains high amounts of phosphate bearing mineral in highly insoluble form.

4 REQUIREMENTS

4.1 Description

The PROM shall be in powdered or granular form, dark brown to black in colour with no foul odour.

4.2 Particle Size

The particle size of PROM shall not be more than 6.0 mm, and a minimum of 90 percent of PROM shall pass through 6.0 mm IS sieve [see IS 460 (Part 1)].

4.3 The product shall also conform to the requirements given in Table 1.

Table 1 Requirements for Phosphate Rich Organic Manure
(Clause 4.3)

SI No.	Characteristic	Requirement	Method of test, Ref to
(1)	(2)	(3)	(4)
i)	Bulk density, g/cm ³ , <i>Max</i>	1.70	Annex A
ii)	Moisture, percent by mass, <i>Max</i>	25	Annex B
iii)	pH	6.5 - 7.5	Annex C
iv)	Conductivity, dsm ⁻¹ , <i>Max</i>	8.0	Annex D
v)	Total organic carbon, percent by mass of total dry mass, <i>Min</i>	7.5	Annex E
vi)	Total phosphorus (as P ₂ O ₅), percent by mass of total dry mass, <i>Min</i>	10.5	Annex F
vii)	Total citrate soluble phosphorus (as P ₂ O ₅), percent by mass of total dry mass, <i>Min</i>	3.5	Annex F

Table 1 (Concluded)

SI No.	Characteristic	Requirement	Method of test, Ref to
(1)	(2)	(3)	(4)
viii)	C : N ratio, <i>Max</i>	20 : 1	Annex G
ix)	Arsenic (as As ₂ O ₃), mg/kg on dry mass basis, <i>Max</i>	10	Annex H
x)	Cadmium (as Cd), mg/kg on dry mass basis, <i>Max</i>	5	Annex J
xi)	Chromium (as Cr), mg/kg on dry mass basis, <i>Max</i>	50	Annex J
xii)	Copper (as Cu), mg/kg on dry mass basis, <i>Max</i>	300	Annex J
xiii)	Lead (as Pb), mg/kg on dry mass basis, <i>Max</i>	100	Annex J
xiv)	Nickel (as Ni), mg/kg on dry weight basis, <i>Max</i>	50	Annex J
xv)	Zinc (as Zn), mg/kg on dry mass basis, <i>Max</i>	1 000	Annex J
xvi)	Mercury (as Hg), mg/kg on dry mass basis, <i>Max</i>	0.15	Annex K

5 PACKING, MARKING AND STORAGE

5.1 Packing

PROM shall be packed in the manner as agreed to between the purchaser and the supplier. Each bag shall be securely closed.

5.2 Marking

5.2.1 Each pack shall be marked legibly and indelibly with the following information:

- Name and type of the material;
- Name, address of the manufacturer;
- Indication of the source of manufacture;
- Percent by mass of total phosphorous to total dry mass;
- Percent by mass of total available phosphorus to total dry mass;
- C : N ratio;
- Gross and net quantity in kg;
- Batch number, in code or otherwise, to enable the lot of manufacture to be traced back from records; and
- Any other information as required under *Fertilizer (Control) Order, 1985* and the *Legal Metrology (Packaged Commodities) Rules, 2011*.

5.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 General Requirements of Sampling

In drawing samples, the following measures and

precautions should be observed:

- Samples shall not be taken at a place exposed to rain/sun.
- The sampling instruments shall be clean and dry when used.
- The material being sampled, the sampling instruments and the bags of samples shall be free from any adventitious contaminations.
- To draw a representative sample, the contents of each bag selected for sampling shall be mixed as thoroughly as possible by suitable means.
- The sample shall be kept in a suitable, clean, dry and airtight glass or screwed hard polythene bottle of about 400 g capacity or in a thick-gauged polyethylene bag. This shall be put in a cloth bag, which shall be sealed after putting inside the detailed description.
- Identifiable details may also be put on the cloth bag like sample no./code no. or any other details which enables its identification.
- Each sample bag shall be sealed air tight after filling and marked with details of sample.

6.2 Sampling from Bagged Material

6.2.1 Scale of Sampling

6.2.1.1 Lot (for manufacturers)

All bags in a single consignment of the material of the same total organic carbon, nitrogen, phosphorous and potassium content and C : N ratio derived from the same source of manufacture drawn from a single batch of the manufacturer shall constitute a lot. If a consignment is declared to consist of different batches of manufacturer, all the bags of each batch shall constitute a separate lot. In the case of a consignment drawn from a continuous process, 2 000 bags (or 100 tonnes) of the material

shall constitute a lot.

6.2.1.2 Lot (for dealer)

The lot is an identifiable quantity of manure with same total organic carbon, nitrogen, phosphorous and potassium content and C : N ratio derived from the same source of manufacture stored at an identifiable place subject to a maximum limit of 100 tones. The lot shall be identified based on visible appearance of bags, their packing and storage conditions. The stock of less than 100 tones with a dealer may also constitute one or more lots, if the material of different sources and brand is available in such quantities.

6.2.1.3 Selection of bags for sampling

The number of bags to be chosen from a lot shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause 6.2)

Sl No.	Lot Size (No. of Bags)	No. of Bags to be Selected for Sampling
(1)	N (2)	n (3)
i)	Upto 10	1
ii)	11 - 100	2
iii)	101 - 200	3
iv)	201 - 400	4
v)	401 - 600	5
vi)	601 - 800	6
vii)	801 - 1 000	7
viii)	1 001 - 1 300	8
ix)	1 301 - 1 600	9
x)	1 601 - 2 000	10

All the bags of a lot should be arranged in a systematic manner. Start counting from any bag randomly; go on counting as 1,2,3,...up to R and so on. R being equal to the integral of N/n . Thus, every 4th bag counted shall be withdrawn and all bags shall constitute the sample bags from where the sample is to be drawn for preparing a composite sample.

6.3 Sampling from Big Godowns/High Stockings

If the procedure given in 6.2.1.3 is not possible to be adopted, the sample should be drawn from the randomly selected compost bags from different layers, from top and from all open sides in a zig-zag fashion.

6.3.1 Sampling from Small Godowns

All the fertilizer bags of the same grade and type of each manufacturer though received on different

dates shall be segregated and properly stacked. All bags of same grade and type of compost manufactured by a particular manufacturing unit may be considered as one lot based on their physical conditions and the sample shall be drawn as per procedure laid down in 6.2.1.3 and 6.4.

6.4 Sampling Probe

6.4.1 An appropriate sampling instrument to be used for collection of a representative sample is called sampling probe. The probe may comprise of a slotted single tube with solid cone tip made of stainless steel or brass. The length of the probe may be approximately 60 cm to 65 cm and the diameter of the tube may be approximately 1.5 cm and the slot width may be 1.2 cm to 1.3 cm. The probe may be used if the physical condition of the manure and the packing material permits its use.

6.4.2 In case of high-density polythene packing and also when the manure is not in free flowing condition, the use of sampling probe may not be possible. In such case, selected bags for drawing samples may be opened and the fertilizers may be taken out of the bags and spread on a clean surface and samples drawn with the help of a suitable sampling device, which may be made of stainless steel or brass cup.

6.5 Drawal of Samples from Bags

6.5.1 Drawal of Sample and Preparation of Composite Samples

Draw, with an appropriate sampling instrument, (sampling probe) small portions of the material from the selected bags as per procedure in 6.2.1.2 and 6.2.1.3. The sampling probe shall be inserted in the bag from one corner to another diagonally and when filled with fertilizer, the probe is withdrawn and fertilizer is emptied in a container/ or on polythene sheet/or on a clean hard surface and made into one composite sample.

6.5.2 If the bags do not permit the use of sampling probe, empty the contents of the bags on a level, clean and hard surface and draw a composite sample by the process of quartering as described under 6.7.

6.6 Quantity of One Sample

One sample of PROM shall have approximately 500 g.

6.7 Preparation of Composite Sample

If the composite sample collected from the different selected bags is larger than required quantity, its size shall be reduced by method of quartering. For quartering, spread the composite sample on a level,

clean, hard surface, flatten it out and divide it into four equal parts. Remove any diagonally opposite parts. Mix the two remaining parts together to form a cone, flatten out the cone and repeat the operation of quartering till a composite sample of required quantity is obtained.

6.8 Preparation of Test Sample and Reference Sample

6.8.1 The composite sample obtained above shall be spread out on a clean, hard surface and divided into three approximately equal portions each of the weight as specified in **6.6**. Each of these samples shall constitute the test sample.

6.8.2 Each test sample shall be immediately transferred to a suitable container as defined under

6.1 e). The slip with detailed description may be put inside the sample bag. Each bag shall also be properly labeled as mentioned in **5.2**.

6.8.3 Each test sample container shall then be sealed.

7 TESTS

7.1 Tests shall be carried out by the methods prescribed in **4**.

7.2 Unless otherwise specified, quality reagents, chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect experimental results.

ANNEX A

[Table 1, Sl No. (i)]

ESTIMATION OF BULK DENSITY**A-1 APPARATUS**

Weighing balance, measuring cylinder (100 ml), rubber pad (0.093 m²; 2.57 cm thickness), hot air oven.

A-2 PROCEDURE

Dry the PROM sample in a hot air oven at 60 °C for 24 hours. Weigh a dry 100 ml cylinder. Fill the cylinder with the sample up to the 100 ml mark. Weigh the cylinder along with the sample. Tap the cylinder for two min for compacting the sample. Measure the compact volume.

A-3 CALCULATION

$$\text{Bulk density, (g/cm}^3\text{)} = \frac{W_2 - W_1}{V}$$

where

W_1 = Mass, in g, of the dry 100 ml cylinder

W_2 = Mass, in g, of the dry 100 ml cylinder with sample

V = Final volume, in ml, up to which the sample is present in the cylinder after compaction

ANNEX B

[Table 1, Sl No. (ii)]

ESTIMATION OF MOISTURE CONTENT**B-1 APPARATUS**

Petri dish, hot air oven, desiccator, weighing balance.

B-2 PROCEDURE

Weigh a clean dry petri dish. Take approximately (weigh to the nearest mg) 10 g of the prepared sample in the petri dish. Heat in an oven for about 24 hours at 70 °C to constant weight. Cool in a desiccator and weigh. Report percentage loss in weight as moisture content.

B-3 CALCULATION

$$\text{Moisture percent by mass} = \frac{100 (B - C)}{B - A}$$

where

A = Mass, in g, of the petri dish

B = Mass, in g, of the petri dish with sample, before drying

C = Mass, in g, of the petri dish with sample, after drying

ANNEX C

[Table 1, Sl No. (iii)]

ESTIMATION OF pH

C-1 REAGENT — Distilled water

C-2 APPARATUS — Rotary shaker, Whatman no. 1 or equivalent filter paper, Buchner funnel fitted with vacuum pump, pH meter.

C-3 PROCEDURE

Make 25 g of PROM into a suspension in 50 ml of

distilled water and shake on a rotary shaker for 2 hours. Filter through Whatman no. 1 or equivalent filter paper under vacuum using a Buchner funnel. Determine pH of the filtrate by pH meter.

ANNEX D

[Table 1, Sl No. (iv)]

ESTIMATION OF CONDUCTIVITY

D-1 REAGENTS — Potassium chloride (AR grade), distilled water.

D-2 APPARATUS — Conductivity meter (with temperature compensation system), 4 mm sieve, flask (250 ml), funnel (outer diameter = 75 mm), beaker (100 ml), analytical balance, filter paper.

D-3 PROCEDURE

Pass fresh sample of PROM through a 4 mm sieve.

Take 20 g of the sample and add 100 ml of distilled water to it to give a ratio of 1:5. Stir for an hour at regular intervals. Calibrate the conductivity meter by using 0.01 M potassium chloride solution. Measure the conductivity of the unfiltered compost suspension. Express the results as dsm^{-1} at 25 °C specifying the dilution of the compost suspension, that is, 1 : 5.

ANNEX E

[Table 1, Sl No. (v)]

ESTIMATION OF TOTAL ORGANIC CARBON**E-1 APPARATUS**

Silica/platinum crucible (60 ml capacity), muffle furnace, analytical balance, hot air oven, desiccator.

E-2 PROCEDURE

Accurately weigh 10 g oven dried sample (70 °C for 24 hours) in to a pre-weighed crucible and ignite the material in a muffle furnace at 550 °C for 4 hours. Cool to room temperature and keep in desiccator for 12 hours. Weigh the contents with crucible.

E-3 CALCULATION

Calculate the total organic carbon by the following formulae:

Total organic matter (percent by mass of total dry mass)

$$= \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

where

W_1 = Mass, in g, of the empty crucible

W_2 = Mass, in g, of the crucible containing oven-dry compost sample

W_3 = Mass, in g, of the crucible containing ash (after combustion)

Total organic carbon (percent by mass of total dry mass) =

$$\frac{\text{Total organic matter (percent by mass of total dry mass)}}{1.724}$$

ANNEX F

[Table 1, Sl No. (vi) and (vii)]

ESTIMATION OF TOTAL PHOSPHOROUS AND TOTAL CITRATE SOLUBLE PHOSPHORUS

F-1 PREPARATION OF SAMPLE FOR ESTIMATION OF TOTAL PHOSPHORUS**F-1.1 Reagent**

Concentrated nitric acid, 70 percent to 72 percent perchloric acid, distilled water

F-1.2 Apparatus

Conical flasks, kjeldahl long neck flasks (500 ml capacity), beaker (100 ml), Whatman no.1 filter paper, hotplate, volumetric flask (100 ml)

F-1.3 Procedure

Accurately weigh 1 g oven dried sample (at 70 °C for 24 hours) in a long neck Kjeldahl digestion flask; add 20 ml to 30 ml concentrated nitric acid and boil gently for 30 min to 45 min. Cool and add 10 ml to 20 ml of 70 percent to 72 percent perchloric acid. Boil gently till fuming point until solution is colorless or nearly so for about an hour. Do not boil to dryness at any time. Cool slightly and add 50 ml water and boil for few min. Cool and filter. Transfer the filtrate to 100 ml volumetric flask and make up the volume to 100 ml.

F-2 PREPARATION OF SAMPLE FOR ESTIMATION OF TOTAL CITRATE SOLUBLE PHOSPHORUS**F-2.1 Reagent****F-2.1.1 Ammonium Citrate Solution**

Dissolve 300 g pure citric acid in 1 500 ml distilled water. Add approximately 345 ml of 28 percent to 29 percent of ammonium hydroxide so than acid is neutralized and a pH of 7.0 is attained. pH can be adjusted by adding ammonium hydroxide or citric acid solutions. This can be stored up to 6 months. Frequently check pH, if changed make it neutral.

F-2.2 Apparatus

Conical flasks, volumetric flasks, water bath, Buchner funnel, vacuum pump.

F- 2.3 Procedure

F-2.3.1 Take 1 g of accurately weighed oven dried sample in a conical flask and add 100 ml of neutral ammonium citrate solution previously heated to 65 °C over water bath.

F-2.3.2 Stopper the flask with rubber cork and shake vigorously for 10 min. Release pressure by opening cork occasionally.

F-2.3.3 Heat the flask on water bath at 65 °C for about an hour.

F-2.3.4 Filter the contents using filter pump/vacuum pump (through Whatman no. 5 filter paper or Buchner funnel) and wash with about 50 ml of distilled water. Transfer the filtrate to 200 ml volumetric flask and make up the volume to 200 ml.

F-2.3.5 Take suitable aliquot of this extract and estimate P by gravimetric method as described below.

F-3 GRAVIMETRIC QUINOLINE METHOD FOR DETERMINATION OF TOTAL PHOSPHORUS**F-3.1 Reagents****F-3.1.1 Citric Molybdic Acid Reagent**

Dissolve 54 g, 100 percent molybdic anhydride (MoO_3) and 12 g NaOH with stirring in 400 ml hot water and cool. Dissolve 60 g citric acid in mixture of 140 ml HCl and 300 ml water and cool. Gradually add molybdic solution to citric acid solution with stirring. Cool, filter and dilute to 1 litre (solution may be green or blue colour depending on exposure to light). If necessary, add 0.5 percent KBrO_3 solution drop by drop until green colour becomes pale. Store in dark in polyethylene bottle.

F-3.1.2 Quinoline Solution

Dissolve 50 ml synthetic quinoline with stirring in mixture of 60 ml HCl and 300 ml water. Cool dilute to 1 litre and filter. Store in polyethylene bottle.

F-3.1.3 Quimociac Reagent

Dissolve 70 g of sodium molybdate dehydrate in 150 ml water. Dissolve 60 g citric acid in mixture of 85 ml HNO_3 and 150 ml water and cool. Gradually add molybdate solution to citric acid-nitric acid mixture with stirring. Dissolve 5 ml synthetic quinoline in mixture of 35 ml HNO_3 and 100 ml water. Gradually add this solution to molybdate-citric-nitric acid solution mix and let it stand for 24 hours. Filter, add 280 ml acetone, dilute to 1 litre with water and mix well. Store in polyethylene bottle.

F-3.2 Apparatus

Erlenmeyer flask, pipette, watch glass, hot plate, sintered glass gooch crucible Grade 4 (30 ml capacity), desiccators.

F-3.3 Procedure

In 500 ml Erlenmeyer flask, pipette aliquot containing not more than 25 mg P_2O_5 (as prepared above by the methods described at **F-1** and **F-2**), dilute to approximately 100 ml with water. Proceed with one of the following method.

F-3.3.1 Add 30 ml citric-molybdic acid reagent and boil gently for 3 min (solution must be precipitate free at this stage). Remove from heat and swirl carefully. Immediately add from burette 10 ml quinoline solution with continuous swirling (add first 3 ml to 4 ml drop wise and remainder in steady stream).

F-3.3.2 Add 50 ml quimociac reagent, cover with watch glass place on hot plate in well ventilated hood and boil for 1 min.

After treatment with **F-3.3.1** or **F-3.3.2**, cool to room temperature, swirl carefully 3-4 times during cooling, filterthrough sintered glass Gooch crucible Grade 4 (30 ml capacity), previously dried at 250 °C and weighed, and wash 5 times with 25 ml portion of water. Dry the crucible and contents for 30 min at 250 °C. Cool in desiccator to constant weight as quinoline phosphomolybdate $[(C_9H_7N)_3H_3PO_4 \cdot 12MoO_3]$. Estimate blank reading without sample. Subtract blank weight. Multiply by 0.032 07 to obtain weight of P_2O_5 . Report as percent P_2O_5 .

F-3.4 Calculation

Total phosphorus (as P_2O_5), percent by mass of total dry mass:

$$= (A - B) \times 0.032\ 07$$

where

A = Mass, in g, of quinoline phosphomolybdate obtained by treatment of sample ; and

B = Mass, in g, of quinoline phosphomolybdate in blank treatment.

ANNEX G

[Table 1, Sl No. (viii)]

CALCULATION OF C : N RATIO**G-1 PROCEDURE**

The C : N ratio is calculated using total organic carbon, percent by mass of total dry mass determined at **E-3** and total nitrogen (as N), percent by mass of total dry mass determined at **G-2.4**.

G-1.1 CALCULATION

C : N ratio = $A : B$

where

A = total organic carbon, percent by mass of total dry mass determined at **E-3**; and

B = total nitrogen (as N), percent by mass of total dry mass determined at **G-2.4**

G-2 ESTIMATION OF TOTAL NITROGEN**G-2.1 Apparatus**

G-2.1.1 Suitable Kjeldahl assembly consisting of 500 ml to 800 ml round bottom, digestion flask and kjeldahl distillation assembly consisting of 500 ml

to 800 ml distillation flask, splash head tube and condenser, all with appropriate glass joints. The length of the condenser's delivery tube should be long enough to keep immersed in a flask for ammonia absorption.

G-2.1.2 Kjeldahl digestion unit with heating control, suitable for 500 ml to 800 ml flasks.

G-2.2 REAGENTS

G-2.2.1 Sulphuric Acid, 93-98 Percent, N-free.

G-2.2.2 Salicylic Acid, reagent grade, N-free.

G-2.2.3 Sodium Thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$), reagent grade.

G-2.2.4 Zinc Dust, Impalpable Powder.

G-2.2.5 Copper Sulphate

G-2.2.6 Potassium or Sodium Sulphate

G-2.2.7 45 percent NaOH Solution

Dissolve 450 gm of Sodium hydroxide pellets in

distilled water and makeup the volume to 1 000 ml.

G-2.2.8 Methyl Red Indicator

Dissolve 1 gm methyl red in 200 ml alcohol.

G-2.2.9 Hydrochloric or Sulphuric Acid Standard Solution, 0.1 N or as per requirement.

G-2.2.10 Sodium Hydroxide Standard Solution, 0.1 N or as per Requirement.

G-2.3 PROCEDURE

Place weighed sample (0.7 g to 2.2 g) in digestion flask. Add 40 ml H_2SO_4 containing 2 g salicylic acid. Shake until thoroughly mixed and let stand, with occasional shaking, 30 min or more. Then add 5 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ or 2 g zinc dust (as impalpable powder not granulated zinc or filing). Shake the flask and let it stand for five min then heat over low flame until frothing ceases.

Turn off heat, add 0.7 g copper sulphate, 15 g powdered K_2SO_4 (or anhydrous Na_2SO_4) and boil briskly until solution clears, continue boiling for another at least 2 hour. Remove from burner and cool, add 200 ml of water and swirl the flask to dissolve all the contents. Transfer to 500 ml volumetric flask, giving several washings with water to the digestion flask. Make up the volume to 500 ml.

Take 25 ml aliquot in the distillation flask; add 300 ml water and a pinch of zinc dust. Take 20 ml of standard acid solution in the receiving conical flask, add 4 drops of methyl red indicator and keep the flask at the lower end of the condenser in such a way that the lower tip of the condenser is fully immersed in acid solution.

Add 30 ml of 45 percent NaOH to the distillation flask, gently so that the contents do not mix. Immediately connect the flask to distillation assembly and swirl to mix the contents. Heat until all the ammonia is distilled (at least 150 ml distillate). Remove from receiving flask. Rinse outlet tube into receiving flask with a small amount of distilled water.

Titrate the contents in the receiver conical flask with standard NaOH solution. Determine blank on reagents using same quantity of standard acid in receiving conical flask.

G-2.4 CALCULATION

Total nitrogen (as N), percent by mass of total dry mass

$$= \frac{1.401 (V_1 N_1 - V_2 N_2) - (V_3 N_1 - V_4 N_2) df}{W}$$

where

V_1 = Volume, in ml, of standard acid taken in receiver flask for sample;

V_2 = Volume, in ml, of standard NaOH used in titrating standard acid in receiver flask after distillation of test sample;

V_3 = Volume, in ml, of standard acid taken in receiver flask for blank;

V_4 = Volume, in ml, of standard NaOH used in titrating standard acid in receiver flask after distillation in blank;

N_1 = Normality of standard acid;

N_2 = Normality of standard NaOH;

W = Mass, in g, of sample taken; and

df = Dilution factor of sample.

ANNEX H

[Table 1, Sl No. (ix)]

ESTIMATION OF ARSENIC**H-1 APPARATUS**

Analytical balance, beaker, hot plate, Whatman no.1 filter paper, volumetric flask (100 ml), atomic absorption spectrophotometer attached with vapour generation assembly.

H-2 REAGENTS

Aquaregia ($\text{HNO}_3 + \text{HCl}$ in a ratio of 1 : 3), concentrated hydrochloric acid, double distilled water, potassium iodide, standard arsenic solution.

H-3 PROCEDURE**H-3.1 Processing of Sample**

Suspend 10 g finely ground sample in 30 ml aquaregia ($\text{HNO}_3 + \text{HCl}$ in a ratio of 1 : 3) in a beaker. Keep on hot plate till moist black residue is obtained (do not dry). Add 5 ml aquaregia and allow to dry on hot plate till residue is moist. Dissolve the residue in 30 ml concentrated HCl and filter through

Whatman no.1 filter paper in 100 ml volumetric flask. Wash filter paper 3 - 4 times with double distilled water. Make up the volume to 100 ml. Take 1 ml of this solution in 100 ml volumetric flask, add 5ml concentrated hydrochloric acid and 2 g potassium iodide and make up the volume to 100 ml. Prepare standards having concentrations of 0.05 ppm, 0.1 ppm and 0.2 ppm by diluting 0.05 ml, 0.1 ml and 0.2 ml of standard arsenic solution with double distilled water in a volumetric flask by making up the volume to 100 ml.

H-3.2 Measurement

Estimate arsenic using vapour generation assembly attached to atomic absorption spectrophotometer as per the method given for instrument.

H-3.3 Expression of Result

Express the metal concentration as mg/kg on oven dry mass basis.

ANNEX J

[Table 1, Sl No. (x) to (xv)]

ESTIMATION OF CADMIUM, COPPER, CHROMIUM, LEAD, NICKEL AND ZINC**J-1 REAGENTS****J-1.1 Diacid Mixture**

Mix 9 parts of nitric acid (HNO_3) and 4 parts of perchloric acid (HClO_4).

J-1.2 Triacid Mixture

Mix 10 parts of nitric acid (HNO_3), 1 part of sulphuric acid (H_2SO_4) and 4 parts of Perchloric acid (HClO_4).

J-1.3 Standard Cadmium Solution**J-1.4 Standard Copper Solution****J-1.5 Standard Chromium Solution****J-1.6 Standard Lead Solution****J-1.7 Standard Nickel Solution****J-1.8 Standard Zinc Solution****J-1.9 Double Distilled Water****J-2 APPARATUS**

Analytical balance, hot air oven, 0.2 mm sieve, conical flask (250 ml), glass funnel, hot plate, Whatman filter paper no. 42, atomic absorption spectrophotometer, volumetric flasks (100 ml)

J-3 PROCEDURE**J-3.1 Processing of Sample**

Take 1.0 g or suitable quantity of oven dried (70°C for 24 hours) sample thoroughly ground and sieved through 0.2 mm sieve in a conical flask. Add 30 ml triacid mixture (use diacid mixture for lead determination), cover it up with a small glass funnel for refluxing. Digest the sample at 200°C on a hot plat till the volume is significantly reduced with a whitish residue. After cooling, filter the sample through Whatman no. 42 filter paper. Make up the volume to 100 ml with double distilled water in a volumetric flask.

J-3.2 Preparation of Working Standards

Dilute 1, 2, 3 and 4 ml of standard 100 ppm cadmium, copper, chromium, lead, nickel and zinc standard solution with double distilled water in separate volumetric flasks and makeup the volumes to 100 ml to obtain standards having concentrations of 1, 2, 3 and 4 ppm.

J-3.3 Measurement

Estimate the metal concentrations of cadmium,

copper, chromium, lead, nickel and zinc by flaming the standards and samples using atomic absorption spectrophotometer (AAS) as per the method given for instrument at recommended wavelength for each element. Run a blank following the same procedure.

J-3.4 Expression of Result

Express the metal concentration as mg/kg on dry mass basis.

ANNEX K

[Table 1, Sl No. (xvi)]

ESTIMATION OF MERCURY**K-1 REAGENTS****K-1.1 Concentrated Nitric Acid (HNO₃)****K-1.2 Concentrated Sulphuric Acid (H₂SO₄)****K-1.3 Potassium Persulphate (5 percent Solution)**

Dissolve 50g of K₂S₂O₈ in 1 litre of distilled water.

K-1.4 Potassium Permanganate (5 percent Solution)

Dissolve 50 g of KMnO₄ in 1 litre of distilled water.

K-1.5 Hydroxylamine Sodium Chloride Solution

Dissolve 120 g of Hydroxylamine salt and 120 g of sodium chloride (NaCl) in 1 litre of distilled water.

K-1.6 Stannous Chloride (20 percent)

Dissolve 20 g of SnCl₂ in 100 ml distilled water.

K-1.7 Standard Mercury Solution**K-1.8 Double Distilled Water****K-2 APPARATUS**

Water bath, flameless atomic absorption spectrophotometer (AAS attached with vapour generation assembly that is, cold vapour mercury analyzer), BOD bottle (300 ml), analytical balance, hot air oven, volumetric flask (100 ml).

K-3 PROCEDURE**K-3.1 Processing of Sample**

Take 5 g (finely ground but not dried) sample in oven at temperature of 70 °C for 24 hour for moisture estimation. Take another 5 g sample (finely ground but not dried) in a BOD bottle, add to it 2.5 ml of concentrated HNO₃, 5 ml of concentrated H₂SO₄ and 15 ml of 5 percent KMnO₄. After 15 min add 8 ml of 5 percent K₂S₂O₈. Close the bottle with the lid and digest it on the water bath at 95 °C for 2 hour. After cooling to room temperature add 5 ml hydroxylamine sodium chloride solution.

K-3.2 Preparation of Standard

Prepare standards having concentrations of 0.05 ppm, 0.1 ppm and 0.2 ppm by diluting 0.05 ml, 0.1 ml and 0.2 ml of standard arsenic solution with double distilled water in a volumetric flask and make up the volume to 100 ml.

K-3.3 Measurement

Reduction of digested sample is brought out with 5 ml of 20 percent SnCl₂ immediately before taking the readings. Estimate mercury concentration using a cold vapour mercury analyzer or atomic absorption spectrophotometry as per the method given for instrument. run a blank following the same procedure.

K-3.4 Expression of Results

Express the mercury concentration as mg/ kg on oven dry mass basis.

ANNEX L
(Foreword)

COMMITTEE COMPOSITION

Soil Quality and Fertilizers Sectional committee, FAD 07

<i>Organization</i>	<i>Representative(s)</i>
Rajmata Vijayaraje Scindia Krishi Vishwa Vidyalaya	DR A. K. SINGH (Chairperson)
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National Bureau of Agriculturally Important Microorganism (NBAIM), Mau Nath Bhanjan	DR ANIL KUMAR SAXENA DR ALOK KUMAR SRIVASTAVA (<i>Alternate</i>)

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